

PCT

INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>5</sup> :</b> <b>A61K 7/34, 7/38</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 91/08732</b> <b>(43) International Publication Date:</b> 27 June 1991 (27.06.91)
<b>(21) International Application Number:</b> PCT/US90/07100 <b>(22) International Filing Date:</b> 4 December 1990 (04.12.90) <b>(30) Priority data:</b> 447,622 8 December 1989 (08.12.89) US 618,704 27 November 1990 (27.11.90) US <b>(71) Applicant:</b> THE GILLETTE COMPANY [US/US]; Prudential Tower Building, Boston, MA 02199 (US). <b>(72) Inventor:</b> KWASS, Jill, Alison ; 10 Porter Road, Andover, MA 01810 (US). <b>(74) Agents:</b> HANDELMAN, Joseph, H. et al.; Ladas & Parry, 26 West 61 Street, New York, NY 10023 (US).		<b>(81) Designated States:</b> AT (European patent), AU, BE (European patent), BR, CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FI, FR (European patent), GB (European patent), GR (European patent), IT (European patent), LU (European patent), NL (European patent), NO, SE (European patent).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> ANTIPERSPIRANT  <b>(57) Abstract</b> <p>An optically clear liquid antiperspirant product in the form of a stable water-in-oil emulsion with a viscosity of less than about 1000 cps at about room temperature includes an aqueous phase with an antiperspirant active ingredient in solution therein, an oil phase making up at least about thirty percent of the product, and a stabilizing agent that has a substantial solubility in each of the oil and aqueous phases. The emulsion has long term stability over temperature ranges from about 0 to 45 °C. The product preferably is dispensed as a thin film from a rollon type dispenser.</p>		

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FI	Finland	ML	Mali
AU	Australia	FR	France	MN	Mongolia
BB	Barbados	GA	Gabon	MR	Mauritania
BE	Belgium	GB	United Kingdom	MW	Malawi
BF	Burkina Faso	CN	Guinea	NL	Netherlands
BG	Bulgaria	GR	Greece	NO	Norway
BJ	Benin	HU	Hungary	PL	Poland
BR	Brazil	IT	Italy	RO	Romania
CA	Canada	JP	Japan	SD	Sudan
CF	Central African Republic	KP	Democratic People's Republic of Korea	SE	Sweden
CG	Congo			SN	Senegal
CH	Switzerland	KR	Republic of Korea	SU	Soviet Union
CI	Côte d'Ivoire	LI	Liechtenstein	TD	Chad
CM	Cameroon	LK	Sri Lanka	TG	Togo
DE	Germany	LU	Luxembourg	US	United States of America
DK	Denmark	MC	Monaco		
ES	Spain	MG	Madagascar		

ANTIPERSPIRANT

This invention relates to antiperspirant products and processes for forming antiperspirant products.

5           Antiperspirant products are well-known in the cosmetic art and typical antiperspirants contain an active antiperspirant ingredient and a vehicle. The active antiperspirant ingredient acts to reduce perspiration, it is believed, by interacting in solution  
10 with sweat glands. Antiperspirant products may be in the form of a dispersion, solution or suspension, e.g., a solid suspension such as a stick or a solid-liquid suspension such as an aerosol or a roll-on. The product may also be an emulsion, which is a stable, homogeneous  
15 mixture of immiscible liquids such as an aqueous phase and an oil phase. Antiperspirant emulsion products may have a range of viscosities from a free standing gel that is used by rubbing an area of the body such as the underarm to a liquid form emulsion that may be applied  
20 as a roll-on to apply a layer to the skin. It is desirable that all antiperspirant products have aesthetic characteristics of smoothness, non-oiliness and non-tackiness. Another desirable characteristic is that no readily visible residue as, e.g., a white layer,  
25 be on the skin after an antiperspirant is applied. It is also important that the product efficiently introduce the antiperspirant active ingredient to the sweat glands.

In accordance with one aspect of the

- 2 -

invention, there is provided an efficacious liquid antiperspirant product that is clear, washable, leaves no visually perceptible residue, and dries quickly. The product, unlike a gel which typically includes a colloidal suspension in a coagulated condition, is a stable, free-flowing liquid water-in-oil emulsion with a viscosity of less than about 1000 cps at about room temperature. The emulsion includes an aqueous phase with an antiperspirant active ingredient in solution therein, an oil phase making up at least about thirty percent of the product and a stabilizing agent. The stabilizing agent has a substantial solubility in the aqueous and oil phases and stabilizes the emulsion in the temperature range from about 0 to 45°C. without impairing the room temperature clarity of the product. The product produces a thin film when applied to the skin and has a use range from about room to body temperature. The product preferably has a viscosity of about 500-900 cps at room temperature and may be dispensed from a roll-on type dispenser. Percentages given herein are in weight percent.

An optically clear antiperspirant product of the invention is one that is visually clear, with a minimal amount of haziness or cloudiness. Like glass, the product allows ready viewing of objects behind it. by contrast, a translucent antiperspirant product, although allowing light to pass through, causes the light to be so scattered that it will be impossible to see clearly objects behind the translucent product. Preferably, the product has a refractive index (measured at 5893Å) of 1.39 - 1.42 at 21°C., and an optical clarity better than one hundred NTU (Nephelometric Turbidity Units) at 21°C. and is packaged in a container of the roll-on type that has an optically clear wall. The turbidity measurements discussed hereinafter were made with a Orbeco-Hellig #965 Direct-Reading Turbidimeter.

- 3 -

Preferably the product is stable for at least a period of three months. A stable emulsion, as discussed herein, is one that does not phase-separate into distinct oil and water portions over time and under normal temperature conditions of use. The stabilizing agent is a bridging component that has a significant solubility in the aqueous phase, and enhances stability of the emulsion at low viscosities suitable for roll-on application. Additionally, the stabilizing agent does not impair the clarity of the product. The stabilizing agent preferably includes a polyalkoxylated alcohol and a lower alcohol such as ethanol, methanol or propanol that solubilizes the polyalkoxylated alcohol, the lower alcohol having a molecular weight less than the polyalkoxylated alcohol. The polyalkoxylated alcohol also improves the washability of the product since the oil phase of the emulsion is generally insoluble in water. Particular polyalkoxylated alcohols are Oleth-5 and PPG-10 Butanediol. Preferably the stabilizing agent is soluble at five percent or more in the oil and water phases.

The oil phase preferably makes up more than about thirty percent of the product and includes an emulsifier which when properly mixed with the polar components yields a water-in-oil emulsion. The oil phase preferably comprises one or a combination of polyether substituted water-in-oil silicone emulsifiers such as cyclomethicone and dimethicone copolyol, dimethicone, and cyclomethicone. Volatile silicones, for example, cyclomethicones such as D-4, DC 244 or DC 344 may be used to enhance the dryness of application. Other silicones such as dimethicones, for example, DC-200, may be employed as a detackifier. (All above available from Dow Corning). A particular emulsifier is a polyether substituted silicone such as cyclomethicone and dimethicone copolyol (available as DC 3225C from Dow Corning).

- 4 -

The aqueous phase includes one or a combination of polar species such as water and propylene glycol and an antiperspirant active ingredient in solution. Preferred active antiperspirant components for use in the antiperspirant products include well-known salts of aluminum chloride, aluminum zirconium chlorohydrates, polyhydroxy complexes of basic aluminum salts, and such salts buffered, for example, with glycine or a polyglycol. Propylene glycol may be added for emolliency and for refractive index adjustment. A multipurpose adjunct, such as ethanol, may also be added for refractive index modification, aesthetic properties and antimicrobial activity.

Products of the invention also provide essentially complete absence of discernible whitening, and high antiperspirant activity. The liquid product, a substantially clear emulsion with a significant proportion of water, has reduced whitening effect as the antiperspirant active is maintained in solution. At the same time, the dissolved antiperspirant ingredient can interact with the sweat glands for effective sweat reduction.

A particular antiperspirant product has an oil phase of about 28 to 30% volatile silicone, about 8 to 10% silicone emulsifier and about 1 to 2% nonvolatile silicone; an aqueous phase of about 25 to 40% water, about 3 to 5% propylene glycol and about 15 to 20% antiperspirant active; and a stabilizing agent that includes about 0.5 to 1.5% polyalkoxylated alcohol and about 4 to 15% lower alcohol.

Preferably, the refractive indices of the aqueous and oil phases are substantially matched and the refractive index of the product is between about 1.39 to 1.42. The product can also contain additional ancillary ingredients such as emollients, colorants, fragrances, and preservatives such as antioxidants. When the stabilizing agent includes Ol th-5, it is preferred to

- 5 -

use an antioxidant such as Tocopherol (e.g. C vi-Ox T-70 (Henk 1)) in an amount of about 0.2% of the amount of Oleth-5.

Products of the invention are made by  
5 preparing an emulsion with a viscosity of less than about 1000 cps at room temperature. From an aqueous phase including an antiperspirant active in solution therein, an oil phase and a stabilizing agent having at least five percent solubility in the aqueous phase. The  
10 aqueous phase, the oil phase, and the stabilizing agent are mixed such that the oil phase makes up at least thirty percent of the product and the mixture is processed to produce a stable emulsion with a viscosity of less than about 1000 cps at room temperature (21°C.).  
15 Preferably, a fragrance is solubilized in the stabilizing phase and then combined with the aqueous phase. The mixture of the stabilizing phase and the aqueous phase is then added to the oil phase as the oil phase is being sheared in an in-line device to form a  
20 stable emulsion of viscosity of under 1000 cps at room temperature.

The following Examples 1-4 are given by way of illustration only and are not to be considered as being limiting. Example 5 is outside the scope of the  
25 invention and is included for comparison purposes. The amounts in the Examples and the claims are in weight percent.

In each of the following Examples, about thirty kilograms of product is made. The ingredients of  
30 the oil phase, the aqueous phase and the stabilizing agent are formulated. The combined stabilizing phase and aqueous phase are then added to the oil phase at room temperature and are sheared in a continuous in-line high shear device such as a Gifford-Wood tandem shear  
35 pipeline mixer or an IKA Dispax-Reactor to produce the desired emulsion with a viscosity of less than about 1000 cps at room temperature.

- 6 -

Example 1

	<u>CFTA NAME</u>	<u>% ACTIVE</u>
	<u>Oil Phase</u>	
5	CYCLOMETHICONE & DIMETHICONE COPOLYOL (DC 3225C)	8.63
	CYCLOMETHICONE (D-4)	28.48
	DIMETHICONE (DC-200 50 cs.)	1.72
	<u>Aqueous Phase</u>	
10	WATER	32.50
	PROPYLENE GLYCOL	4.27
	ALUMINUM ZIRCONIUM TETRACHLOROHYDREX-GLY	17.50
	<u>Stabilizing Agent</u>	
	OLETH-5 (EMULGIN 05)	1.00
15	ETHANOL (SD ALCOHOL 40, 200 PROOF)	5.90
		100.00

The resulting composition of Example 1 had a viscosity of 800 cps, a measured turbidity of 31.0 NTU and remained stable for at least three months.

Example 2

	<u>CFTA NAME</u>	<u>% ACTIVE</u>
	<u>Oil Phase</u>	
20	CYCLOMETHICONE & DIMETHICONE COPOLYOL (DC 3225C)	8.63
	CYCLOMETHICONE (D-4)	28.33
25	DIMETHICONE (DC-200 50 cs.)	1.72
	<u>Aqueous Phase</u>	
	WATER	28.23
	PROPYLENE GLYCOL	4.34
	ALUMINUM ZIRCONIUM TETRACHLOROHYDREX-GLY	15.20
30	<u>Stabilizing Agent</u>	
	OLETH-5 (EMULGIN 05)	1.04
	ETHANOL (SD ALCOHOL 40, 200 PROOF)	12.36
	FRAGRANCE	0.15
		100.00

The resulting composition of Example 2 had a measured turbidity of about 25 NTU.



- 7 -

Example 3

	<u>CFTA NAME</u>	<u>% ACTIVE</u>
	<u>Oil Phase</u>	
5	CYCLOMETHICONE & DIMETHICONE COPOLYOL (DC 3225C)	8.63
	CYCLOMETHICONE (D-4)	28.48
	DIMETHICONE (DC-200 50 cs.)	1.72
	<u>Aqueous Phase</u>	
	WATER	
10	PROPYLENE GLYCOL	32.50
	ALUMINUM ZIRCONIUM TETRACHLOROHYDREX-GLY	4.27
		17.50
	<u>Stabilizing Agent</u>	
	PPG-10 BUTANEDIOL (MACOL 57)	1.00
15	ETHANOL (SD ALCOHOL 40, 200 PROOF)	5.90
		100.00

The resulting composition of Example 3 had a viscosity of 520 cps, a refractive index of 1.3970, a measured turbidity of about 25 NTU and remained stable for at least three months.

20

Example 4

	<u>CFTA NAME</u>	<u>% ACTIVE</u>
	<u>Oil Phase</u>	
	CYCLOMETHICONE & DIMETHICONE COPOLYOL (DC 3225C)	
25	CYCLOMETHICONE (D-4)	8.63
	DIMETHICONE (DC-200 50 cs.)	28.48
		1.72
	<u>Aqueous Phase</u>	
	WATER	
30	PROPYLENE GLYCOL	32.50
	ALUMINUM ZIRCONIUM TETRACHLOROHYDREX-GLY	4.27
		17.50
	<u>Stabilizing Agent</u>	
	OLETH-5 (EMULGIN 05)	0.50
	PPG-10 BUTANEDIOL (MACOL 57)	0.50
35	ETHANOL (SD ALCOHOL 40, 200 PROOF)	5.90
		100.00

The resulting composition of Example 4 had a viscosity of about 800 cps, a measured turbidity of 26 NTU and remained stable for at least three months.

- 8 -

Example 5

	<u>CFTA NAME</u>	<u>% ACTIVE</u>
	<u>Oil Phase</u>	
5	VOLATILE SILICONE (DC 244)	33.00
	CYCLOMETHICONE-DIMETHICONE COPOLYOL (DC 3225C)	10.00
	VOLATILE SILICONE (DC 200)	2.00
	<u>Aqueous Phase</u>	
10	ALUMINUM ZIRCONIUM TETRACHLOROHYDRATE-GLY (35%) SOLN.	50.00
	PROPYLENE GLYCOL	<u>5.00</u>
		100.00

After processing, the formulation of Example 5 produced a product having a viscosity of 800 cps, and a turbidity of 11.6 NTU. However, this Example produced an unstable emulsion that separates and is therefore commercially unsuitable as an antiperspirant roll-on product.

While particular embodiments of the invention has been shown and described, various modifications will be apparent to those skilled in the art, and therefore it is not intended that the invention be limited to the disclosed embodiments or to details thereof, and departures may be made therefrom within the spirit and scope of the invention.

- 9 -

C L A I M S

1. A substantially clear antiperspirant product comprising:
  - a stable water-in-oil emulsion with a viscosity of less than about 1000 cps at about room temperature, having:
    - (a) an aqueous phase with an antiperspirant active ingredient in solution therein;
    - (b) an oil phase making up at least about thirty percent of said product; and
    - (c) a stabilizing agent having a substantial solubility in both said oil phase and said aqueous phase to stabilize said emulsion in the temperature range from about 0 to 45°C. without impairing the clarity of said product in a product use temperature range from about room to body temperature.
2. The product of claim 1, wherein said viscosity is about 500-900 cps at about room temperature.
3. The product of claim 1 or 2, including a roll-on type dispenser with an optically clear wall in which said product is housed for dispensing.
4. The product of any one of the preceding claims, wherein said emulsion is stable for at least a period of three months.
5. The product of any one of the preceding claims, wherein said stabilizing agent includes a polyalkoxylated alcohol and a lower alcohol having a molecular weight less than polyalkoxylated alcohol.
6. The product of claim 5, wherein said polyalkoxylated alcohol is Oleth-5 or PPG-10 Butanediol.
7. The product of any one of the preceding claims, wherein said stabilizing agent is soluble at five percent or more in each of said oil and aqueous phases.
8. The product of any one of the preceding claims, wherein the refractive index of said product is between about 1.39 to 1.42.
9. The product of claim 8, wherein its optical

- 10 -

clarity at room temperature is better than 100 NTU.

10. The product of any one of the preceding claims, wherein said oil phase comprises about 28 to 30 percent volatile silicone, about 1 to 2 percent nonvolatile silicone and about 8 to 10 percent silicone emulsifier; said aqueous phase comprises about 25 to 40 percent water, about 3 to 5 percent propylene glycol and about 15 to 20 percent antiperspirant active; and said stabilizing agent comprises about 0.5 to 1.5 percent polyalkoxylated alcohol and about 4 to 15 percent lower alcohol.

11. The product of any one of the preceding claims, wherein said oil phase includes one or a combination of polyether substituted silicone water-in-oil emulsifiers such as cyclomethicone and dimethicone copolyol, dimethicone, and cyclomethicone.

12. The product of any one of the preceding claims, wherein said aqueous phase is one or a combination of water, glycol, glycol derivatives, and/or alcohols.

13. The product of any one of the preceding claims, wherein said antiperspirant active ingredient is aluminum zirconium chlorohydrate, aluminum chlorohydrate, or polyhydroxy complexes of basic aluminum salts, or such salts buffered, for example, with glycine or a polyglycol.

14. The product of any one of the preceding claims, which includes a fragrance.

15. A method of preparing a substantially clear antiperspirant product, comprising preparing an emulsion with a viscosity of less than about 1000 cps at room temperature by

(a) providing an aqueous phase including an antiperspirant active ingredient incorporated therein;

(b) providing an oil phase;

(c) providing a stabilizing agent having a substantial solubility in said aqueous phase of said product; and

(d) mixing said aqueous phase, oil phase, and

- 11 -

stabilizing agent such that said oil phase makes up at least thirty percent of said product to produce a stable, clear emulsion having viscosity of less than about 1000 cps at room temperature and a product use temperature range from about room to body temperatures.

16. The method of claim 15, including adding a fragrance to said stabilizing agent prior to mixing said phases.

17. The method of claim 15 or 16, including adding an antioxidant.

18. The method of any one of claims 15 to 17, wherein said stabilizing agent includes a polyalkoxylated alcohol and a lower alcohol that has a molecular weight less than said polyalkoxylated alcohol.

19. The method of claim 18, wherein said polyalkoxylated alcohol is Oleth-5 or PPG-10 Butanediol, and said lower alcohol is ethanol, methanol or propanol.

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US90/07100

**I. CLASSIFICATION F SUBJECT MATTER** (if several classification symbols apply, indicate all) <sup>3</sup>

According to International Patent Classification (IPC) or to both National Classification and IPC

INT CL(5): A61K 7/34 A61K 7/38

ILS CL: 424/66 424/68

**II. FIELDS SEARCHED**

Minimum Documentation Searched \*

Classification System :

Classification Symbols

U.S. 424/66

424/68

Documentation Searched other than Minimum Documentation  
to the Extent that such Documents are Included in the Fields Searched \*

**III. DOCUMENTS CONSIDERED TO BE RELEVANT <sup>11</sup>**

Category *	Citation of Document, <sup>14</sup> with indication, where appropriate, of the relevant passages <sup>17</sup>	Relevant to Claim No. <sup>15</sup>
X	US, A, 4,122,029 (GEEETAL) 24 October 1978 See column 9, line 44 to 59	1 to 19
X	US, A, 4,725,431 (HOURIHAN ET AL) 16 February 1988 See column 1, lines 29 to 50	1 to 19
X	US, A, 4,782,095 (GAM) 01 November 1988 See column 14, lines 23 to 32	1 to 19
X	Cosmetics & toiletries, A December, 1985, page 65 to 75, column 4, lines 20 to 38.	1 to 19

\* Special categories of cited documents: <sup>16</sup>

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

**IV. CERTIFICATION**

Date of the Actual Completion of the International Search :

11 MARCH 1991

Date of Mailing of this International Search Report :

04 APR 1991

International Searching Authority :

ISA/USA

Signature of Authorized Officer <sup>20</sup>

DALE R. ORE